BORON FILAMENT MANUFACTURE IN SPACE — A LITERATURE FEASIBILITY STUDY

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ABSTRACT

This paper presents a comprehensive review of technical literature pertaining to the manufacture of boron filament on Earth. A possible space manufacturing process for boron filament is described. Several central substrate deposition techniques, using different core materials, electric decomposition, and other boron-containing raw materials, are discussed. Nonsubstrate deposition techniques, such as glow discharge on a rotating disc or between two pointed tungsten electrodes, are described. Several techniques to manufacture boron filament from the melt are discussed. These include point destruction of surface tension, electrical discharge forming, melt drawing, Czochralski crystal growth, the Taylor process, drawing a substrate through a melt, and melt extrusion. This survey points out a way to manufacture boron filament in space that involves heating high-purity boron to 2800°F in boron nitride crucibles, and then transferring it into an RF-positioning field for further heating by induction coils or hot gas.

I. INTRODUCTION

NASA is presently attempting to simulate interest in private industry for their idea of manufacturing goods in space for eventual use on Earth, utilizing the unique environment available in Earth orbit of near-zero gravity, huge vacuum, and nearly infinite black body.[1-5] Although the concept is now a few years old at NASA, its first extensive presentation to industry was at a 1968 conference[2] attended by invited heads of various American companies, including President Lew Evans of Grumman. At the end of this conference, at which NASA

summarized the possible uses of the space environment for manufacturing, a call went out for private companies to identify and develop areas of potential cost saving to themselves by manufacturing in space. This report summarizes one of the two areas considered by Grumman to hold some promise for our own development.

The use of zero-gravity must be the central focus of any manufacturing process designed for the space factory. In general, this limits uniqueness considerations to the liquid state, although certain gaseous state processes might be significantly improved in space. One specific category of liquid processes that might be considered is the processing of highly corrosive or high melting point materials, such as boron, which could be held by RF fields without crucible contact in the liquid state. Boron melts at 3755 $\pm 30^{o}$F and is extremely corrosive above this temperature. There is no known crucible material that will not erode or corrode in contact with this liquid metal.

Filaments of boron are in great demand at this time primarily for composite filler material. By far the cheapest method of manufacturing filaments (Figure 1) of any substance is to draw from the melt. Highly viscous materials, such as glasses and plastics are presently manufactured in this way. Unfortunately, the rheology of molten metals and many other materials has so far disallowed this process. As a result, filaments of these materials must be manufactured by more expensive means. Materials with a reasonable amount of ductility can be solid extruded through ever smaller dies. Others, including boron, must be formed electrochemically or by chemical vapor deposition (CVD) on a substrate.[6]

It would be highly desirable for boron filaments to be fabricable from the melt. Not only could cost be reduced to a level competitive with graphite in the future, at about $30 to $50 per pound, but the filament itself might be improved in strength from the 400,000 psi of present material to well over a million psi.[7] In addition, rates of production could be increased a hundred to a thousandfold, smaller diameter filaments would be economically possible, and boron composites would be used in various nuclear applications for which they are presently restricted by the tungsten core of boron filaments. In order to do this, however, a method of holding and shaping the boron into filaments without touching a crucible must be developed. Several possibilities can be considered for this, but the best appears to involve holding the boron mass in a high-frequency coil arrangement under zero-gravity conditions in space. Filaments might then be formed from the mass by: Czochralski crystal growing, filament drawing, breaking the surface tension with a point heat source, electroforming, or melt extrusion through a field die.

II. PRESENT METHOD OF MANUFACTURE OF BORON FILAMENTS

A. Structure

Commercial quantities of continuous boron filaments are today made by chemical vapor deposition on a 0.0005-inch diameter hot tungsten filament from BCl_3 gas.[8-19] This process has been described by many writers, and its exact details are unimportant here (Figure 2). The filaments so produced range in diameter from 0.003 to 0.005 inch and in lengths up to 10,000 feet or more.

During the course of deposition, the tungsten core generally disappears entirely, being replaced by reacted tungsten borides, mostly W_2B_5 and WB_4 (Figure 3). The cylindrical boride core thus formed in the filament center is only 0.00065 inch in diameter, and shows a very distinct interface with the bulk boron due to the much higher diffusivity of boron in tungsten than vice versa. Very little tungsten manages to cross this boundary into the bulk boron. The interface area is full of small voids produced by a Kirkendall vacancy condensation effect after rapid boron diffusion into the core.

The surface of CVD-produced boron filaments has a characteristic noduled or grainy appearance. These nodules grow larger as the filament diameter increases up to 0.0035 inch, but become smaller beyond this diameter. Their absolute size varies from run to run. The cause of this nodule effect has been found to be die marks on the surface of the original tungsten filament. The die marks provide sites of preferred nucleation and growth for the boron, which grows in cones outward. It is highly important, therefore, for the substrate material to be as smooth as possible.

In addition to nodules, the filament surface occasionally contains inclusions, most probably picked up at the mercury contact seal at the exit of the reaction chamber. Electron microprobe analysis has shown that these inclusions contain Mg, Cl, Cr, and Ca.

The bulk boron has a crystal structure, called "amorphous" which has been identified in X-ray, electron diffraction, and grain grown growth studies[20] as a microcrystalline β-rhombohedral structure. There is some controversey over the size of these crystallites. X-ray studies place them at approximately 20 Å. Some electron-diffraction investigations confirm the 20 Å value. However, other electron-diffraction studies indicate a highly strained, oriented, layered platelet structure with crystallites of 2000 Å size.

High residual stresses are known to occur in CVD-produced boron filaments. The core is in a compression of 150,000 to 200,000 psi, the surface in compression at 70,000 psi, and the first deposited layers in tension at 120,000 psi. The primary cause of these stresses appears to be the increase in core size as the filament forms, although other factors, such as temperature fluctuations during growth, thermal expansion mismatch between boron and core materials, and quenching at the mercury exit contact seal, also play a part. The stress

pattern apparently varies, greatly from filament to filament. These high stresses cause radial cracks (extending from the compressed interface across the tensed first layers of bulk boron to the outer, compressed layers) to occur in all CVD-produced boron filaments.

B. Properties

CVD-produced boron filaments have mechanical properties highly desirable for composite materials (Figure 4). Tensile strength values average 400,000 psi with a standard deviation of about 125,000 psi, measured for a standard 1-inch gage length; torsion shear strength values are about 500,000 psi. The Young's modulus ranges consistently from 55 to 60 x 10^6 psi, while the modulus of rigidity is measured at 26 x 10^6 psi. The Poisson's ratio is 0.21.

The density of boron is very low. Bulk material has a density of 2.34 g/cm^3 (compared to 2.70 for aluminum and 1.8 for beryllium), and the tungsten boride core of a 0.004-inch diameter filament only increases this value to 2.6 g/cm^3. Its specific tensile strength value is therefore 4.0 x 10^6 inches. Comparisons show these to be the best combination of filament properties available for composites. However, despite these high values, tests have shown they are actually quite low compared to what is possible for boron.

There are basically four major sites for fracture initiation in CVD-produced filaments (Figure 5). The temperature range necessary to obtain the "amorphous" boron is somewhat narrow. As a result large crystals of β-rhombohedral boron are formed occasionally and these markedly decrease strength values to below 200,000 psi.

At somewhat higher strength values - between 200,000 and 400,000 psi - large inclusions, due to impure deposition conditions, initiate fracture. These inclusions range in size from 2.5 to 20μ; the strength effect of particles smaller than these is masked by other factors.

The third strength-limiting site, again applicable in the range 200,000 to 400,000 psi, is the noduled surface of the CVD filaments. Under the right deposition conditions this is eliminated in as-produced filaments; alternatively, chemical polishing, removing approximately 0.005 inch from the surface, will eliminate the nodule effect. In general, chemical polishing is necessary to eliminate this strength-limiter.

Bend-test and other evidence has shown the existence of a fourth strength limitation site to be the core at the 400,000 to 500,000 psi level. Weibull has shown theoretically and experimentally that the bend-to-tensile strength ratio for a rectangular homogeneous material is 1.41. Despite a difference in test geometry, the same ratio for as-produced boron filaments is about 1.6. However, chemically polished filaments without surface flaws show a ratio of 3.8, much too high for accord with the theory. The explanation for this

discrepancy lies with the filament core. In the tensile case, the stress is applied uniformly over the cross-section so that any flaw present in the specimen will concentrate stresses and cause failure. However, in the bend case, the central plane of the filament (the core area) experiences no stress, while the surface is stressed to a maximum. Thus, the core area limits strength in the tensile tests, but not in the bend tests, causing the high 3.8 ratio.

The bend test indicates the true stress levels possible to bulk boron, if the core could be eliminated. That strength has been conservatively calculated as 26×10^5 psi in tension and 39×10^5 psi in bending. These calculated values, obtained from the measured modulus of elasticity of CVD-produced filaments, are probably too low, since higher moduli up to 70×10^6 psi have been measured on micro-crystalline boron[21] In fact values of flexural strength exceeding 2×10^6, and as high as 2.7×10^6 psi, have been obtained on chemically polished specimens. According to the Weibull ratio, therefore, pure boron filaments should easily give tensile strengths of 5/8 of 2×10^6 psi, or at least 1.25×10^6 psi. (Actually these values should be higher since the core has a finite size in small filaments and will reduce bend test values to some extent.)

There is further direct evidence that the core area limits strength. In tests on split boron filaments, with an average tensile strength of 250,000 psi, the core was etched out. These filaments then presented average strengths of 650,000 psi, with high values of 890,000 psi. And finally fractography studies confirm its role as a stress limiter.

There are several reasons for the tungsten/tungsten-boride interface to act as a stress concentration.[20] First, the two materials have markedly different mechanical and physical properties. Second, the high compression/tension stress difference across the interface already discussed adds to any applied stresses. Third, the Kirkendall voids along the core interface act as stress concentrators.

In addition to limiting strength, all four of these fracture sites (i.e., structural irregularities, surface defects, bulk inclusions, and the core interface) produce the great variation in strength values presently obtained on boron filaments.

To what extent can these stress-limiting and stress varying factors be eliminated? With careful processing, all but the core interface problem can be eliminated.[11] It remains as a strength-limiter in all filaments produced by chemical vapor deposition on tungsten.

C. Further problems (Figure 6.)

Strength values are only one of the problems encountered in chemical-vapor-deposited boron filaments. In addition to this, however, are (1) the nature of the tungsten core, which eliminates boron composite as a nuclear material, due to the nuclear properties of tungsten, (2) the cost of boron filaments, (3) the speed of the manufacturing, and (4) the ultimate size of the filaments, limited by the size of the tungsten core.

Boron can be used in nuclear applications as a neutron absorber and shield, having a thermal neutron cross-section of 750 barns. However, the small amount of tungsten in CVD-produced filaments disallows this use. As a result, boron composite material will not be usable in nuclear applications unless the tungsten core can be eliminated. The future potential usage in this application, if the core can be eliminated, has been estimated at 25% of the future aerospace market, which is indeed large.[22]

The present cost of boron filaments produced by chemical vapor deposition on tungsten is at least $251 per pound (in lots of 3000 lbs.)[23] For the time being this figure provides a competitive edge over graphite (at over $300 per pound) and other potential reinforcement materials.[7] Of course, boron does have certain properties which may continue to make it competitive, e.g. higher strength and slightly higher modulus than graphite. However, projected cost values for the 1970's show that, with increasing usage and manufacturing skill, graphite will far outshine boron-on-tungsten in cost as a composite material. Of the $251 figure for boron on tungsten, over $100 is tied up in the tungsten. Processing costs for this size tungsten filaments are not expected to decrease significantly without major technological breakthroughs. Boron-on-tungsten filaments, as a result, will level out in cost at a predicted level of about $145 per pound by 1975. Graphite, on the other hand, is expected to reach $30 per pound by that time (about the cost of bulk boron). Clearly, unless some major change takes place in manufacture of boron filaments, the material will soon run into strong competition from graphite.

The speed of manufacturing boron filaments is about 1 to 10 feet per minute per filament depending upon the length of the chambers. This is much faster than a method such as Czochralski single-crystal growing; however, melt extrusion processes promise rates on the order of 100 to 1000 times greater per filament.[32] This is considered a highly significant difference.

Finally, the tungsten core can only be manufactured to a certain minimum diameter. Both handling problems and production economies mark this limit. If any smaller diameter is desired, a different process will be required to produce the filament.

III. POSSIBLE METHODS AND PRESENT STATUS

A. Other Chemical Vapor Deposition Methods

Several investigators have attempted to chemically vapor deposit boron on hot wire substrates other than tungsten[12, 15] (Figure 7). Though induction and radiant heating have been tried, resistance heating has been most satisfactory to heat the wire and is the only method developed to date. The temperature necessary to decompose BCl_3 and form "amorphous" boron greatly restricts the possible candidates for the hot wire (B^{24}, Rh, Ta, Ti, Mo, and graphite have been used), and tantalum is the only one to date which could conceivably compete with tungsten. However, the tantalum filaments are more expensive than tungsten and cannot withstand the high temperatures as long, so that today tungsten is used exclusively for the hot-wire process.

If diborane, B_2H_6, is used instead of BCl_3, lower deposition temperatures can be used, because of the lower decomposition temperature of the diborane (500 to 750°C).[13, 17, 25, 26] In this case, other substrate materials can be considered in the hot wire apparatus. One such substrate, which was considered promising and was thoroughly investigated, is fused silica. The price of this composite filament would be about $30 per pound, if it could be made with the same properties as the boron-tungsten. However, average strength levels attained are only 200,000 to 300,000 psi, with normal maximums about 350,000 psi. The strength limiters appear to be two-fold: (1) lack of bonding between boron and core evidenced by fracture origin in the interface and by pullout of the core; and (2) low density apparently caused by polymer and gaseous void formation at the temperatures necessary for deposition. Although it was originally though that diborane would provide faster deposition rates than BCl_3, the deposition has been found to be diffusion limited and maximum rates without detrimental effects on filament strength are no more than are possible with BCl_3.

Chemical vapor deposition on a substrate can also be accomplished by electrical rather than thermal decomposition of a boron-compound gas (Figure 8). This process has the distinct advantage of allowing plating on conductive or nonconductive filaments, since the filament can be a passive receiver surface (although in some configurations it is used as an electrode). These electrical discharge methods are presently in development, but there are many design problems to be overcome including electrode fouling and discharge plasma and field instability. No examples of quality boron filaments produced by these methods are known and they are relatively slow - about 0.8 feet per minute.

All of the processes mentioned so far require a central substrate filament for deposition. This remains as a weak link and/or high cost item in the system. Several systems have been proposed which would eliminate this in CVD-produced filaments.

One of these uses a glow discharge to deposit a thin line of boron on the outside of a rotating disc. This is peeled off to form a continuous filament without a central substrate. Generally, this process suffers from the same problems as normal glow discharge deposition on a substrate, namely electrode and chamber surface foul-ups and instability in the discharge plasma. The filaments obtained are unsymmetrical in cross-section, and initial start-up is very difficult.

Fortunately there is a better means of obtaining substrateless filaments.[27] If a DC glow discharge is ignited between two pointed tungsten electrodes in an atmosphere of BCl_3 and H_2, a boron deposit of filament characteristic forms on the anode and a boron stub of non-filament characteristic forms on the cathode. By withdrawing the anode from the cathode slowly, maintaining a constant discharge gap, short length filaments can be drawn which are limited by the electrical resistance of the filament and by the friable nature of the stub. The process can be made continuous by supplying electricity to the filament at a point near the glow discharge by means of a secondary discharge or a

mercury contact and by continual removal of the cathodic boron deposit. No success has been achieved in attempts to produce more than one filament from the same cathode because of electrical interference between glow discharges.

The gaseous conversion in this process is primarily thermal rather than electrical; temperatures range from 1700°C in the discharge to 1400°C at the cathode to 900°C at the anode. The rate-controlling step was found to be the diffusion rate through the glow discharge surface. One of the major problems with this process results from this — the maximum filament formation rate is only 0.1 feet per minute, which cannot begin to compete with even the CVD on tungsten process. Other major problems or disadvantages of this process are highly critical parameters, such as: difficulty of maintaining clear chambers (as in all glow discharge deposition processes); high fixed-capital and production costs; limitation in diameter of filaments to less than 0.0026 inch; low average strength of filaments of 300,000 to 400,000 psi and large variations in strength values (from well under 300,000 up to 800,000 psi), with little chance for improvement; and, finally, warping of the filaments.

In summary, then, it can be stated that none of the alternative CVD processes, with or without substrate, can compete in the foreseeable future with the deposition on tungsten route. Three of the major disadvantages of the latter — namely, high cost, low strength, and slow production rate — are unlikely to be improved upon, and in fact, would probably be worse, in the alternate processes, even if they were perfected.

B. Melt Processes

As indicated before, there are alternate methods to manufacture filaments other than CVD. Mechanical extrusion cannot be considered for boron because of its high hardness[28] and lack of ductility.[13] In fact, under strain of 400 μ in/in and stress of 30,000 psi in continuous load-unload cycles, polycrystalline boron displays no mechanical hysteresis (perfect elasticity) at a strain sensitivity of 2 μ in/in.[29] And in hardness boron is next to diamond among the known elements with a Mohs scale reading greater than 9. Of the other two possibilities, very little work has been performed on electrochemical boron filament manufacture, which leaves the melt processes for consideration.

There are at least seven distinct possibilities for boron filament manufacture from the melt in various stages of development:

- Point destruction of surface tension
- Electrical discharge forming
- Melt drawing
- Czochralski crystal growth

- Taylor process
- Drawing of substrate through the melt
- Melt extrusion.

The first two processes in this list have not been attempted as far as we know (Figure 9). Point destruction of surface tension was suggested by Dr. C. Lee of Grumman's Research Department.[30] If the surface tension on a liquid is broken at a point, material from the liquid mass will shoot out perpendicular to the surface. One way of breaking the surface tension is to superheat locally, e.g., with an electron beam or continuous laser. Under proper conditions, the material ejected from underneath the interrupted surface might shoot out as a continuous filament. Because no known work has been performed on this subject and even the workability of the basic principles of the process are in some doubt, no further consideration was given to it.

If current is passed through a wire, a cylindrical magnetic force field will form around the wire with the field vector tangential to the wire circumference, and the force vector radially inward. If two parallel wires with currents running in the same directions are placed in close proximity, the interaction of their force fields will tend to attract them to each other. A current-carrying rod may be considered as a bundle of wires with parallel currents. Because of the attraction of parallel currents, the field of this rod will tend to collapse it. While in the solid state, the rigidity of the rod will maintain its shape under high current levels, conducting liquids or gases (plasma) will constrict with a high enough current. Although no known work has been performed on liquids, a great deal of effort has been expended on plasma constriction.[31] Many instability problems are encountered with the plasmas, and there could likewise be expected to present problems in the liquid.

Metals and many other materials cannot in general be drawn from a melt, as is done with glass, because of low viscosity, and boron is probably no exception. Certainly at temperatures at or above the listed melting temperature, the viscosity of 99% pure boron, at only 41 to 50 centipoise, is much too low for either upward or downward drawing. The latter requires a viscosity of around 3000 to 4000 poise (estimated for glass) and a small variation of viscosity with temperature.[33] Boron evidently does have a much higher viscosity just below the melting temperature, sufficient, according to Aerojet-General, to blow bubbles.[32] This requires about 1000 poise[33]; however, the reproducibility of this result is in great doubt. First the existence of a melting range designates an impurity content in the material. Duplication of this impurity content ($\ll 1\%$) and maintenance of the very narrow temperature range at which this viscosity occurred would be improbable at best. In conclusion there is little or no prospect of being able to draw boron from a melt.

In the Czochralski crystal growth process[24, 34-37] (Figure 10), a seed crystal attached to a rod is used to nucleate crystalline material from a melt. The seed is gradually pulled out of the liquid as the crystal forms producing a rod. This process has been used to manufacture pure crystalline rods of boron. However, the rods grown are of large diameter, must be zone refined for proper shape and structure, and seem to be limited in length to about 3 inches. The process is very slow (about 0.5 inches per hour) and holds little promise for continuous filament production.

In the Taylor process[38] molten metal is poured into a hollow tube, usually glass. The tube is then softened, pulled, and cooled, leaving a filament inside. Although the process has been used to manufacture long, extremely smooth, very high-strength filaments of various metals, its applicability to boron composite filaments is doubtful. First, it is difficult to imagine continuous filament production; second, the process is extremely costly; and third, no material is known that can withstand corrosion from liquid boron and could be used as a tube. Therefore, the Taylor process can be eliminated from consideration for boron filament production.

As an intermediate process, utilizing the advantages of speed of the melt process and ease of the tungsten substrate process, an attempt has been made to draw tungsten and silica filaments through molten boron to form a boron filament.[39]

The coatings obtained were highly nonuniform, very thin, and irregularly grained. The tungsten tended to dissolve in the boron, making a tungsten/tungsten-boride filament, instead of one primarily of boron, and contaminating the liquid boron source. Strength was very low, with a maximum of 76,000 psi and average of 58,100 psi; the reasons for this were not examined in detail. Even if rapidly manufactured filaments were obtained, they would retain at least two of the four disadvantages of the CVD boron-on-tungsten process.

IV. MELT EXTRUSION PROCESS

A. Work by Aerojet-General

One of the melt processes, melt extrusion or melt spinning, has shown considerable promise in work performed under Air Force sponsorship by Aerojet-General and Monsanto.[33] Filaments are formed in this process by forcing liquid boron held in a crucible through a tiny orifice using inert gas pressure and solidifying it in a catch chamber below (Figure 11).

Aerojet investigated three basic areas: (1) molten-boron properties pertinent to fiberization, (2) materials for the heating and containment of boron, and (3) techniques and operational parameters related to fiberization. Since the design of melt-extrusion apparatus depends upon knowing certain fundamental physical properties of molten boron, Aerojet first investigated these. The melting point of their technical-grade boron (98.9% purity) was found to be

3736 ±30°F. Higher purity material (99.5%) melted slightly higher at 3755 ±40°F. A thermal arrest, accompanied by softening of the boron, was noted between 3625 and 3675 °F. Boron could be supercooled by more than 100 °F without freezing, even with alloying additions of up to 13.7 wt% titanium or 30.7 wt% zirconium.

It was thought that a low-melting-point eutectic might occur at about 2 wt% carbon in the boron-carbon phase diagram. Carbon was indeed found to decrease the melting point, but only by 25 to 50 °F (with additions of 0.3 to 0.5 wt%C). Neither titanium nor zirconium additions were found to have any effect on the boron melting temperature up to 13.7 wt% titanium or 21 wt% zirconium, although the volume contraction upon freezing was greater with these two additions than with pure or carbon-doped boron.

The viscosity of liquid boron at or near its melting point was found to be 45 ±5 centipoise for the grade investigated. Just below the melting point, over a very narrow temperature range, a viscosity high enough to allow blowing bubbles of boron existed. The normal viscosity of glass under these conditions is about 1000 poise. Carbon contents of 0.4 to 0.7 wt% increased the viscosity to 85 to 100 centipose; however, additions up to 20.7 wt% zirconium or 13.7 wt% titanium had no effect.

The surface tension of molten boron just above the melting point was found to be 1000 dynes/cm.

Aerojet tested many materials for the crucibles and orifices. Boron attacked MfB_2, TaB_2, ZrB_2, NbB_2, in order from least to greatest attack. None could be used in contact with molten boron because of excessive erosion. The same was true of both tantalum and tungsten. The most satisfactory results were obtained with boron nitride, especially when pretreated. For crucibles, satisfactory curing of BN was obtained with a cycle of 5 hours at 3150 °F plus 15 minutes at 3400 °F in argon at 1 atmosphere, while a satisfactory curing cycle for orifice inserts was 1 hour at 2800°F in vacuum followed by 5 minutes at 3400°F in argon at 1 atmosphere. Born will wet BN when: (1) the temperature is raised above 4000 °F. (2) the BN is not properly pretreated, (3) the BN has a low density structure, or (4) more than 0.75% carbon is added to the melt. Wetting is very undesirable in melt extrusion, since it restricts the smooth flow of liquid through the orifice. Even BN was rather unsatisfactory for both crucibles and orifices. Crucibles lasted a maximum of seven extrusion operations (about 12 seconds and 4 grams average for each extrusion – 84 seconds total), while orifice openings gained in area an average of 20% for each such extrusion. Worse, orifice enlargement varied from 2 to 120% unpredictably. While Aerojet claims that "repeated remelting of boron in BN crucibles does not cause significant pickup of impurities by the melt", the erosion indicated above hardly supports this conclusion. In fact, it is evident that BN is not a satisfactory container material for melt extrusion.

Aerojet's attempts to make fibers from the melt were highly unsatisfactory. The maximum length they attained was 0.150 inch, and most of their tests resulted in either no product or spherical shot. The apparatus is standard for this type of work, consisting basically of a crucible with changeable orifice insert; an RF heating coil with tantalum susceptor; a chamber above the crucible which is pressurized with argon gas; and a 6-foot long helium-filled chamber for solidification of the melt below the orifice. All of the fibers manufactured were of the β-rhombohedral variety. The Weber number, defined by $N_{We} = V^2 \rho d/\sigma$, where V = velocity, ρ = density, d = orifice diameter, and σ = surface tension, was found to be a good indication of fiberizability. Best results were obtained in the N_{We} range from 30 to 60. Added carbon up to 0.75 wt%, zirconium up to 20.7 wt%, and titanium up to 13.7% had no effect on the product. The solidification rate was not increased markedly with helium flow added to the area just below the orifice. No tensile tests were reported.

The poor Aerojet results can be attributed to many factors, but chief among them are the following:

- Short solidification chamber; fibers either could not solidify before hitting the bottom of the chamber or, if solidified, broke against the bottom.
- Inability to maintain a constant jet diameter due to orifice enlargement.
- Poor equipment design, resulting in nonrepeatibility of flowing.
- Crucible erosion.

B. Work by Monsanto Research

Monsanto Research has had much greater success with the melt extrusion process. Their investigation covered six basic areas:

- Define the basic parameters of the process and raw material and process control equipment, requirements.
- Investigate and evaluate the fundamental concepts for improving the behavioral characteristics of the charge material.
- Develop the melt spinning apparatus to the level of a filament forming unit capable of precise control over the appropriate operating parameters.
- Determine the broad range of conditions applicable to the formation of continuous boron filament.

- Determine the physical, mechanical, and chemical characteristics of filaments formed and correlate when applicable to process conditions.

- Design and construct a process unit tailored to investigate initial engineering scale-up factors and to reflect proper automatic control techniques.

A discussion of these areas will provide a state-of-the-art look at the melt-extrusion process.

Monsanto's equipment was basically the same as Aerojet's, consisting of a crucible and removable orifice insert; an upper chamber capable of argon gas pressurization; RF heating coils; a graphite ring susceptor to heat the boron charge to a temperature where direct RF coupling becomes efficient (approximately 2730 ^{o}F.); and a catch chamber below the crucible.

Monsanto's first effort was with a research scale unit, the crucible containing only 3.4 cubic centimeters of charge. A large capacity unit was built near the end of the program, but not extensively tested. Successful fiberization was accomplished with their technical-grade boron (99.5% pure); a series of boron minor alloys containing 1 to 2 wt% Ta, W, Zr, Ti, Nb, and V; and two boron compounds, aluminum boride, AlB_{12}, and yttrium hectoboride, YB_{70}.

The factors affecting filament formation were melt temperature and temperature uniformity (extreme sensitivity), extrusion pressure, and charge and fiber purity. For best results the temperature must be held below 4000^{o}F; the higher the melt temperature the worse the final results were.

Orifice wetting by the liquid boron was found to be highly undesirable for fiberizing. Wettability in turn was found to depend upon crucible material and pretreatment. If boron nitride is to be used, it must first be fired to remove volatile impurities. The best compatibility and contact angle were obtained using hot pressed boron nitride pretreated with ammonia at 2700^{o}F or methanol extracted, followed by prefiring at 4000^{o}F under argon. Preliminary results with isotropic and anisotropic pyrolytic boron nitride showed promise of increased mechanical strength.

Overall, boron nitride, aluminum nitride, and thoria were investigated as crucible materials. Only boron nitride showed any promise. However, even this is not a very satisfactory crucible material, suffering serious erosion. And for orifices it is quite unsatisfactory —a 3-gram charge, forced through an orifice made in the very best material, expanded the opening from 0.008- to 0.009-inch diameter, an area increased of 27%. Results show very much worse results at temperatures above 3900^{o}F and with other materials.

A total of 251 fiberization tests were run on the research scale equipment. Streaming occured in 185 of these and filaments formed 85 times. Once proper settings were established, the process was repeatable. An orifice range from

0.002- to 0.006- inch diameter, extruding pressure from 20 to 80 psi, and melt temperature of 3550 to 4500°F were studied, with filaments 0.0035- to 0.008- inch diameter and 1/4- to 11- inch length resulting, in yields varying from 1 to 90 wt%. Actually, high-speed photographic studies showed lengths of up to 2 or 3 feet being produced, but, upon impact at the collection chamber bottom, these broke into the shorter lengths. A "production-scale" unit was built utilizing 25- to 50- gram charges and a longer collection chamber, but testing was not finished.

Monsanto performed a lengthy investigation of the properties of the filament they obtained from the research-scale extrusion unit. All of the filaments were limited in tensile strength by surface pitting to the 50,000 to 60,000 psi range, with best results up to 89,000 psi. Bending strength was somewhat higher, as expected, ranging from 100,000 to 124,000 psi. Thus, the bending/tensile strength ratio is about 2.0. This is somewhat high, but not unexpected considering the existence of both surface and internal defects, and the wide range of tensile and bend strength values. No Young's modulus data were reported; however, bending moduli in the 70 x 10^6 psi range were measured in simple bend tests, with comparative tests indicating that melt-extruded filaments are stiffer than vapor-deposited boron filaments (measured at 45 x 10^6 psi).

An exhaustive examination was made of the possible reasons for surface pitting, including mushy solidification due to alloying elements; entrainment of solid BN, liquid B_2O_3 or various gases; hydrogen from the ambient NH_3 diffusing into the liquid stream and out at the liquid-solid interface; hot tearing; corrosion from catch chamber gas; and plane-front solidification pitting. Monsanto considers that this last explanation is the predominant cause of the surface defects. According to this, the solid-liquid interface of β-rhombohedral boron is likely to be inclined to the fiber cross-section because of its low symmetry crystallographic structure. The mechanism of nucleation and growth in an impurity-laden liquid leads, under this condition, to a pitted surface. There is, however, some ambiguity in Monsanto's conclusions, since in the summary of their work they claim that pitting is <u>not</u> solidification controlled, but rather due to some kind of corrosion. Although Monsanto eliminated BN from consideration as a solid impurity because they said they had not found it either in the body of the filament or in the pits, there is no work reported to this effect, and there is no place for corroded BN from the crucible and orifice to go except to be carried along with the filament. Therefore, it seems likely that BN is to be a prime cause for surface pitting as an impurity, either in solution or as a solid inclusion.

The basic problem with the strength of boron filaments, however, is not pitting, but grain structure. Much of the strength of CVD-produced boron filaments is due to the extreme fineness of the boron microcrystals. In fact, any macrocrystalline material in these filaments greatly weakens them. The grain size of melt-produced boron filaments is generally large in comparison with filament diameter, but varies greatly along the length. Many crystals extend

across the filament completely, while the size of others averages 1/8 to 1/4 of the filament diameter. Because of the low-symmetry crystal structure of boron, it is believed to be highly anisotropic in thermal and mechanical properties. Thus, large non-uniform grains can be expected to act as stress risers under an applied stress. Large grains, also, will tend to cleave catastrophically once a fracture has been initiated, while small grains will resist the propagation of a small crack.

In addition, a smaller grain size will reduce the pitting phenomenon, if this were due to impurity discharge in plane-front solidification. Smaller grain size would indicate more nucleation sites. If enough of these were present, nucleation would be essentially homogeneous and the inclination of the plane front to the filament would disappear. Therefore, no pits would be formed.

The mode of solidification evidenced by the microstructure of Monsanto's filaments shows that nucleation of the large grains takes place at the filament surface. This is due to the lower temperature of the surface and means that potential nucleation sites inside the filament are being overshadowed by nucleation and growth from the surface. It is evident, therefore, that a change in cooling conditions is needed to allow the internal nucleation sites to be active before the surface site in order for smaller grains to form. More heat should be removed longitudinally and less radially from the filament. Of course, high solidification rates must be maintained to form the small grains.

The most effective method for achieving a fine grain structure in a cast material is by adding alloying elements of the proper kind and amount to induce homogeneous nucleation at sites ahead of the advancing solidification front. There are two ways this can be accomplished: stable particles added to the original melt, or precipitation of stable particles at the liquidus from a homogeneous solution. Monsanto did not consider solid particle addition to the melt feasible for the extrusion process, so no work has been performed on this aspect of the problem. They did, however, perform some work on alloying for nucleation.

Theoretically, the most effective alloy nucleant for boron must form a compound that is stable at the liquidus of the melt either with the boron itself or with an impurity, must have a crystal structure similar to the rhombohedral system, and have a unit cell size near that of boron. Monsanto made a literature search for binary and ternary borides, carbides, and nitrides that might be effective nucleating agents. None with theoretically perfect characteristics was found, so trial and error was employed.

From a series of preliminary melts of boron alloys, several were chosen for melt extrusion because of their fine grain size. The best filament results were obtained with B + 2 wt% W alloy. In some areas a very fine grain size was obtained with this alloy, but other areas showed much larger grains. It is thought that incomplete mixing in the charge material is the explanation of the grain size variations. In general, the results of grain refinement show great

promise. Although it was hoped that alloying would reduce surface pitting, no such effect was noted.

Monsanto further characterized the high-temperature thermal arrest, describing it as a "plastic" creep region occurring at 3670°F.

From this discussion, it is clear that a number of Monsanto's problems are due to the contact between molten boron and any crucible or orifice (Figure 12). The process itself looks highly promising if the many problems associated with the crucible/orifice can be overcome. On the other hand, if the mechanical crucible/orifice cannot be eliminated, there is little prospect of this process becoming feasible for manufacture of boron filaments.

V. RF FIELD EXTRUSION PROCESS

A. General Description

In order to eliminate the problems with the crucibles in the melt extrusion process, it is necessary to support, contain, and extrude molten boron without contact from reactive materials. No solid material has been found suitable; no known liquids would be inert at these temperatures, liquid boron control with liquid streams is out of the question, and inert gas control of the boron would also be virtually impossible.

This leaves electric or magnetic field control. However, neither liquid nor solid boron is magnetic. Electrically, boron is a semiconductor[40-47], which means its conductivity increases with rising temperature (unlike metals). At room temperature, boron has a resistivity of 4×10^{6} ohm-cm; at 3495°F the resistivity has dropped to 5×10^{-2} ohm-cm, a change of 8 orders of magnitude. Addition of alloying elements greatly changes the room temperature values toward greater conductivity, but at high temperatures the values for alloys merge with those for pure boron. Because of the high conductivity at high temperatures of boron, electrostatic field positioning is out of the question. However, RF alternating electric fields can be used. For positioning this involves an arrangement sililar to levitation melting.

In levitation melting[48-55], a charge is placed in a high-frequency alternating electric field, in which it is levitated and melted without contact (Figure 13). From this state it can be "poured" out of the field by reducing the field strength and allowing gravity to pull on it. The field constricts the melt as it pours, forming it into a rod which can be solidified. The idea is to further constrict the poured rod down to filament diameter, thus using non-contracting fields to position and form the boron filaments.

There are severe problems with RF levitation melting and pouring semiconductors on Earth. Levitation must be performed against the gravity force, which limits the size of molten material that can be supported on Earth. In addition, gravity provides a fixed force field pulling on the boron. Finally, heating and levitation force cannot, in general, be independently controlled. It becomes desirable, therefore, for these and other reasons, to consider manufacturing boron under zero-gravity conditions, using RF fields to position and hold the boron, while heating, and extruding through a nozzle-shaped field.

B. Levitation Melting

Levitation of a solid or liquid conductor results from placement in a high-frequency alternating electromagnetic field generated between two coaxial coils with currents circulating in opposite directions. Eddy currents induced in the conductor interact with the field to produce supporting and laterally stabilizing forces. A levitated metal floats in space and usually melts.

Levitation melting on Earth was first proposed in 1923, but was not used until 1952, when its feasibility for crucibleless production of ultra-high purity refractory metals was investigated [51]. Though the process thrived for a short while, the inability to stably levitate more than about 20 grams of the denser metals and the development of other methods to accomplish the original levitation melting objectives has slowed the enthusiasm of many researchers.

Two basic parameters determine the levitation force on a sample – the coil current and the frequency. For materials of moderate electrical conductivity like boron, no significant increase in levitation force results as the frequency is increased at very high frequencies. However, the energy absorbed by the conductor becomes so large that melting results, and the temperature rises beyond the desired level. Maximum levitation force with minimum heating is attained at low frequencies and high field strengths and gradients. The value of the frequency to be used depends on the radius and nature of the metal and the temperature desired. Frequency tuning is not normally available over a very wide band, and temperature control thus becomes difficult at best. For a controlled drop out of a coil, in which the charge size is constantly changing, the difficulty is magnified.

The severity of this problem is great. In vacuum melting, separate controls for current and frequency are required for long duration experiments. It has even been proposed that separate means of heating, such as arc, electron beam, or a high-frequency indiction coil, be used in conjunction with the low-frequency levitation coil. However, boron is a low-conductivity material even up to the melting point, and there is no certainty that low frequencies will provide sufficient levitation force. And the lower the conductivity of a material, the more difficult it becomes to control temperature. As we have seen, however, in the melt extrusion of boron temperature must be controlled very closely. It is known, in addition, that RF melting of boron is not possible from the solid state, because of the low conductivity of the material. Thus, a

susceptor was used in Monsanto's equipment to raise the temperature sufficiently for the conductivity to allow direct RF heating of the boron to take place.

Some consideration has been given to the stability of the molten charge and of the pouring liquid in levitation melting. Evidently the surface tension and density determine whether liquid metals will drip during levitation. The levitation force acts solely off the central axis of the coil center, so that surface tension is the sole force holding up the charge on the vertical axis. If the field is sufficiently weak, the surface tension will be insufficient to hold it up, and it will drip or pour at the axis from the bottom of the charge. Though calculations have been made of the steady state conditions (charge surface tension, density, and radii of curvature) necessary to prevent dripping, no calculations of dynamics conditions have yet been performed to take into account a moving charge or oscillations in the system. However, surface tension forces can be expected to inhibit growth of short-wavelength instabilities in the system.

Coil design is still largely empirical. Small tubing diameter is preferable to provide more turns per unit length of coil. Tubing of oval cross-section can be employed for this reason. In practice, 1/8-inch diameter tubing of 0.020-inch wall thickness is the smallest that can be adequately cooled. Coil shape is critical. The smaller the diameter of the coil, the higher the field strength. Cylindrical coils give maximum levitating force, but require coils of reverse current direction on each end for melt stability. Other configurations tried include pancakes, rings, multilayered turns, and inverted cones. Recently some work has been undertaken to provide a more basic characterization of optimum levitation coil design, but the process is still largely trial and error. Specifically, obtaining a proper coil design to form a nozzle for filament formation will have to be the subject of experimentation. In fact, two researchers in the field of levitation melting have indicated doubt that such a design will be found.

Although at least 38 elements have been successfully levitation melted (Figure 14), no reports have been found indicating boron to be among them. However, semiconductors, such as silicon and germanium, have been levitated, and boron has higher conductivity at high temperatures than either of these two, so there is no reason to believe that any special problem will exist with boron. High vapor pressure in the liquid state has prevented certain elements from being levitatable, but molten boron has a relatively low vapor pressure and no such problem is expected. Extremely high melting temperature has prevented molten levitation of tungsten, but again Monsanto used RF melting in their boron work, so boron should melt in levitation also.

Besides the lack of crucible contact, other advantages exist in levitation melting over other means of processing liquid metals. First, the melt is homogenized very rapidly due to very efficient frictionless stirring. Monsanto believed that major difficulties arose in their alloying experiments due to inhomogeneity of the melt during pouring. Thus the great homogeneity possible

to levitation melts should aid the solidification of alloyed boron filaments. Secondly, very rapid heating and cooling rates are possible in a levitation melt, and homogeneity will therefore be retained in the solidified material. Thirdly, it is possible to remove gases from a metal very rapidly and completely in vacuum melting and therefore to control gas content level.

C. RF Positioning Fields

Dr. R. T. Frost discussed the subject of RF field positioning in space at the November, 1968, Huntsville Conference.[2] The accelerational forces that would be present in a space factory would be from two sources: spacecraft thrust, and astronaut body movement. Since thrusting could be controlled during material processing, only the latter need be considered. Dr. Frost's calculations show that for a 25,000-pound space station the maximum accelerations encountered would be 10^{-3} to 10^{-4} g. Since the translational motion of the spacecraft, due to astronaut movement across a 20-foot compartment, is only about 2 inches, it is probable that the design objective for the factory positioner could be a control acceleration of 10^{-4} g. Though a low frequency would be desirable for maximum control of melt shape, it is probable that high frequencies will be necessary, either for heating or for sufficient levitating force, because of the semiconducting nature of boron. For the same reason, high power will also be necessary. The maximum size of a position-controlled melt in space will be limited by the amplitude of oscillations of the molten mass. These oscillations would be initiated by the position restoring forces and, if large enough, would break the surface tension forces of the molten material. They would damp with time due to the viscosity of the melt. Order-of-magnitude calculations show that the mass limitation due to surface oscillations would allow positioning of 10^3 to 10^4 times as much material in space as can be levitated on Earth. For most materials this would mean positioning control of approximately 10 kilograms. For a 10-cm radius sphere, calculations show that these oscillations would disappear after 1000 seconds; for a sphere of radius 1 to several cm, the damping time would drop to 10 to 100 seconds. These order-of-magnitude figures are believed to be applicable to boron melts.

The best coil design for RF positioning could depend upon many factors. In any case, positioning and extruding coil design will have to be integrated. Possible positioning coil designs would include those used presently in levitation work — basically two-coil positioners — or, for more stability, an arrangement consisting of three pairs of orthogonal coils. The exact coil design necessary would be a major problem to overcome in implementing the proposed program.

D. RF Extrusion Nozzle Field And Heat Sources

Just as an RF field must be used to position molten boron in space, one must likewise be used to act as an extrusion die for filament formation. Any of the coils in the two or six-coil designs might be used for this, since a uniform field strength would exist at the center of each coil, and the liquid boron would

form into a cylinder as it passed through. Alternatively a separate coil arrangement might be used, if the field strength necessary to form filaments is significantly different from that necessary to position the boron in space.

The diameter of the extruded boron filament would depend on several factors, including the viscosity of the boron, the strength of the field, the diameter of the coil, and the speed of the extrusion process.

The force necessary to extrude the boron through the field nozzle could be supplied by increasing the strength of the opposing coil in either the two or six-coil arrangements. If this is not possible, an additional RF coil could be added to supply the extrusion force. Another possibility for this is inert gas pressure, although it would be difficult to control this to provide even heating and proper extrusion rate.

NASA has considered the heat sources that might be used for the AAP experiments[54], and their conclusions are equally applicable to the space factory concept. The sources included induction coil, electron beam, laser, electric arc, hot gas, electrical resistance, solar radiation, and chemical heating methods. Of these the electric arc and electron beam are impractical for the present project; with the former there is danger of contamination of the specimen by the electrode material, with the latter there is direct contamination because of the requirement of grounding the specimen. The other methods are all possible, though the laser is heavy, inefficient, and relatively unreliable for production processes at the present time; solar radiation would require orientation capability to keep the collector aimed at the sun; and chemical heating would be expensive and difficult to control. In conclusion, use of either hot gas or induction coil heating could be recommended for heating the boron mass.

VI. SUMMARY

A. Description of Process

From the preceeding discussion, a fairly complete description of the proposed process to manufacture boron filaments in space can be set forth (Figure 15). In barest outline, high-purity boron, doped as necessary for proper crystallization, is placed in a volume in space, where it is heated, either by hot inert gas or other means, to a temperature of about 2800°F. Up to this temperature the boron may be held in boron nitride crucibles. Then it is placed, still in the solid state, in an RF positioning field for holding, and heated further by either induction coils or hot gas. At the melting point, the boron is forced by the hot gas or by an induction coil to exit through the field forming nozzle. There it is cooled by experimentally determined means and collected on spools.

B. Advantages of Process

The potential advantages of space manufacture of boron filaments (Figure 16) are:

- Lower cost
- Higher quality filaments
- Faster production rates
- More potential uses.

The costs of this process should make boron filaments competitive with graphite, especially if the expected material benefit can be realized. Commercial grade boron (90 to 92% pure) could be obtained at a cost of $14 per pound in 5000-pound lots; this could then be purified fairly simply in space where no crucible contact would be necessary for containment. Alternatively technical grade boron (99.5% pure) could be obtained for $50 per pound in 5000-pound lots and used directly. These costs[55] are likely to decline rather sharply as demand for boron increases. Add to this the transportation costs at $5 to $10 per pound, as predicted by Dr. Von Braun[2], and the equipment, labor, and operating costs of the system. The estimated total should be quite competitive with graphite and well below the cost of present filament.

All of the strength problems associated with the core present in CVD-produced filaments should be eliminated by manufacture from the melt. The noduled surface (caused by the uneven surface of the original tungsten wire), the interface voids and cracks, and the residual stresses and different mechanical properties across the core interface all contribute to lowering the tensile and bending stresses that the present boron fibers can withstand. Assuming that proper grain size and fiber surface can be obtained on the melt-produced filaments in space, average tensile strengths of well over a million psi, and bend strengths of over two million psi should be attainable on a production basis. In addition, preliminary results have shown that melt-produced filaments have higher moduli. And the great scatter in strength values, associated with the core and surface defects, should be eliminated, permitting higher design allowables.

Several other advantages should acrue from use of the proposed process. Filaments of much smaller diameter should be manufacturable, if desired, by elimination of the fixed-size core. Production rates should be attained on the order of 100 to 1000 times greater than presently possible. In addition, boron composites would be usable in nuclear applications.

C. Problem Areas and Suggestions for Future Work

There are several major problem areas associated with the proposed process, and these would have to be investigated on a research level (Figure 17).

- Methods must be developed to crystallize molten boron to obtain a fine, even-grain structure. This involves investigations of cooling rates and methods and nucleation techniques. Much of this could be investigated on Earth, utilizing Earth levitation techniques.

- Levitation characteristics of molten boron must be investigated, including a search for stable coil designs. This knowledge should be used for designing space positioning devices for large masses, after experimenting in space with coil positioners. In conjunction with this, an effort should be expended to attempt to extrude molten boron from a levitation melt in filament-sized streams on Earth. If this proves impossible or when scale-up is necessary, these experiments should be performed in Earth orbit.

- The characteristics of fine-grained, high-quality, boron rods or filaments must be tested when these become available to make certain that the mechanical properties anticipated are actually attained.

- The exact design for a production machine must be drawn up. The proper heat source, extrusion force mechanism, environment, coil designs, etc., must be investigated and chosen.

- Once some of these questions have been answered with more certainty, an analysis of costs will have to be made in order to make certain that continuation of the effort will really be worthwhile.

In conclusion, it is clear that any further effort expended on this project must be of a research nature, and that there are many questions which would have to be answered before a production process could be designed. However, the process itself appears to be promising enough to warrant this further research.

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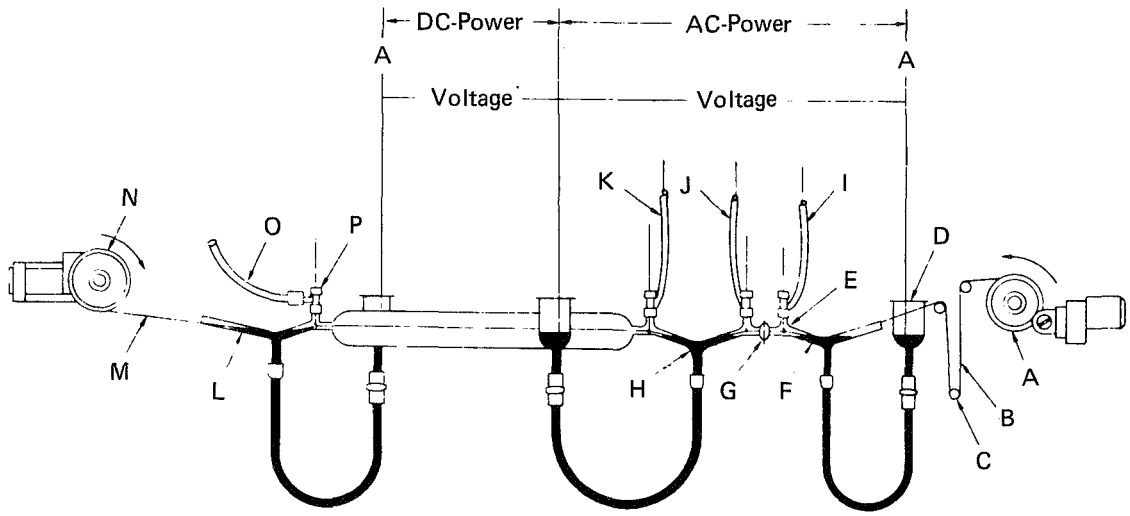
- Mechanical (Wire Drawing, Stretching, Pin Drawing, Broaching, Foil Slitting etc.)
- Electrochemical
- Vapor - Desposition (With Or Without Substrate)
- Liquid - Metal

Figure 1. Methods of Filament Manufacture



A Power-driven substrate feed
B Substrate
C Tension device
D Mercury reservoir
E Guide loop
F Mercury contact seal
G Outgas chamber
I Hydrogen in
J Hydrogen out
K Exhaust gases
L Mercury contact seal
M Boron filament
N Power-driven take-up spool
O Hydrogen and boron trichloride
P T-connector

Figure 2. Apparatus for Continuous Chemical-Vapor-Deposition of Boron on Tungsten From Boron Halide Process[13]

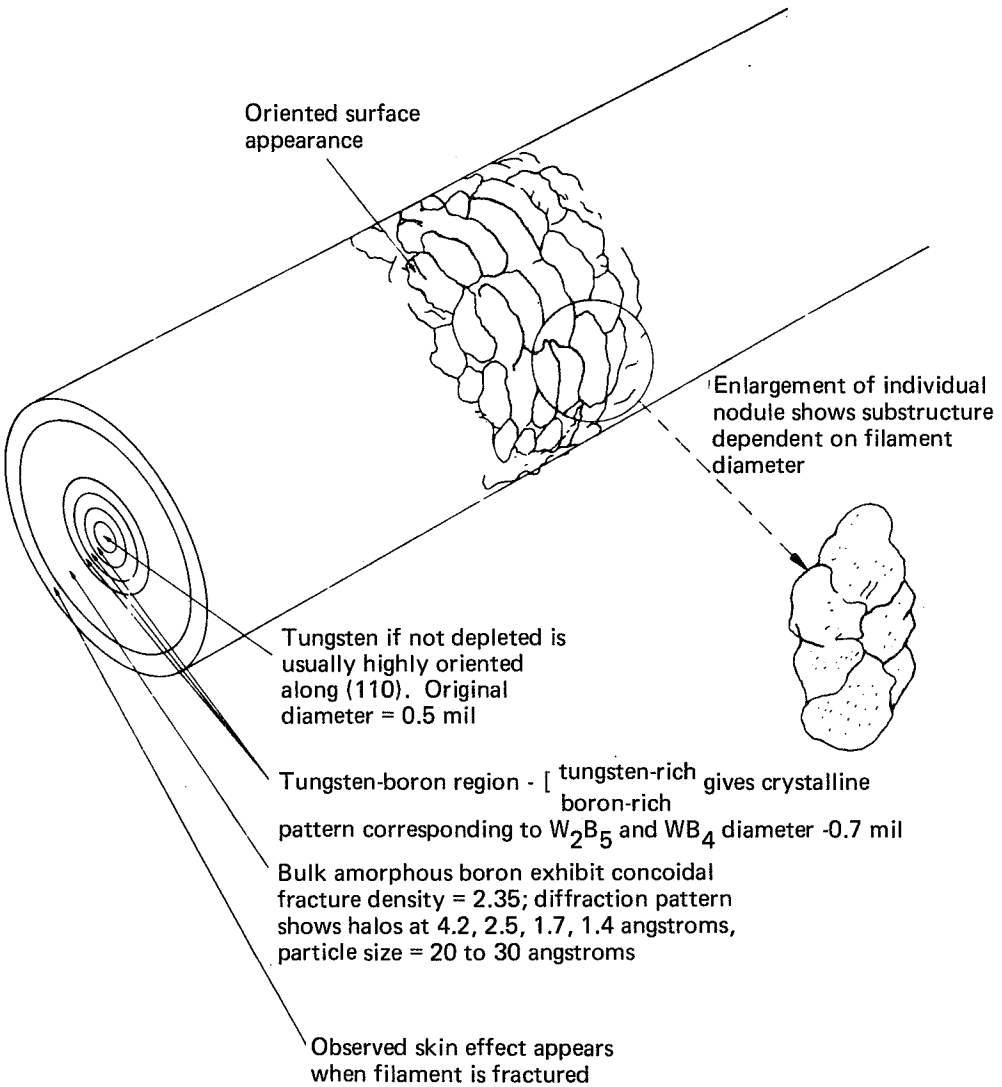

Oriented surface appearance
Enlargement of individual nodule shows substructure dependent on filament diameter
Tungsten if not depleted is usually highly oriented along (110). Original diameter = 0.5 mil
Tungsten-boron region - [tungsten-rich / boron-rich gives crystalline pattern corresponding to W_2B_5 and WB_4 diameter -0.7 mil
Bulk amorphous boron exhibit concoidal fracture density = 2.35; diffraction pattern shows halos at 4.2, 2.5, 1.7, 1.4 angstroms, particle size = 20 to 30 angstroms
Observed skin effect appears when filament is fractured

Figure 3. Structure of CVD-Produced Boron-on-Tungsten Filaments[13]

- High Tensile and Shear Strengths
- High Young's and Rigidity Moduli
- Low Density
- High Specific Strengths and Moduli

} Compared to Other Filament Materials

Figure 4. Properties of CVD-Produced Boron-on-Tungsten Filaments

Site	Tensile Strength Limitation (psi)
Crystals of amorphous boron	less than 200,000
Inclusions of 2.5 to 20 μ size	200,000 to 400,000
Noduled surface	200,000 to 400,000
Core - interface	400,000 to 500,000

Figure 5. Fracture - Initiation Sites

- Low tensile and bending strengths compared to theoretical values
- Low moduli compared to melt-produced filaments
- Large variation of strength values
- Limitation on use in nuclear applications
- High cost
- Low production rate
- Limitation on minimum diameter

Figure 6. Summary of Present Process Disadvantages

A. With Substrate Core

1. Thermal Decomposition
   a. Conductive (W, Ta, B, Rh, Ti, Mo, graphite) or nonconductive (silica) substrates
   b. Resistance, induction, or radiant heating methods
   c. Boron halide or diborane source
2. Electrical Decomposition - Conductive or nonconductive Substrates.

B. Without Substrate

1. Deposition on rotating disc
2. Deposition on anode tip

Figure 7. Summary of Chemical-Vapor-Deposition Processes

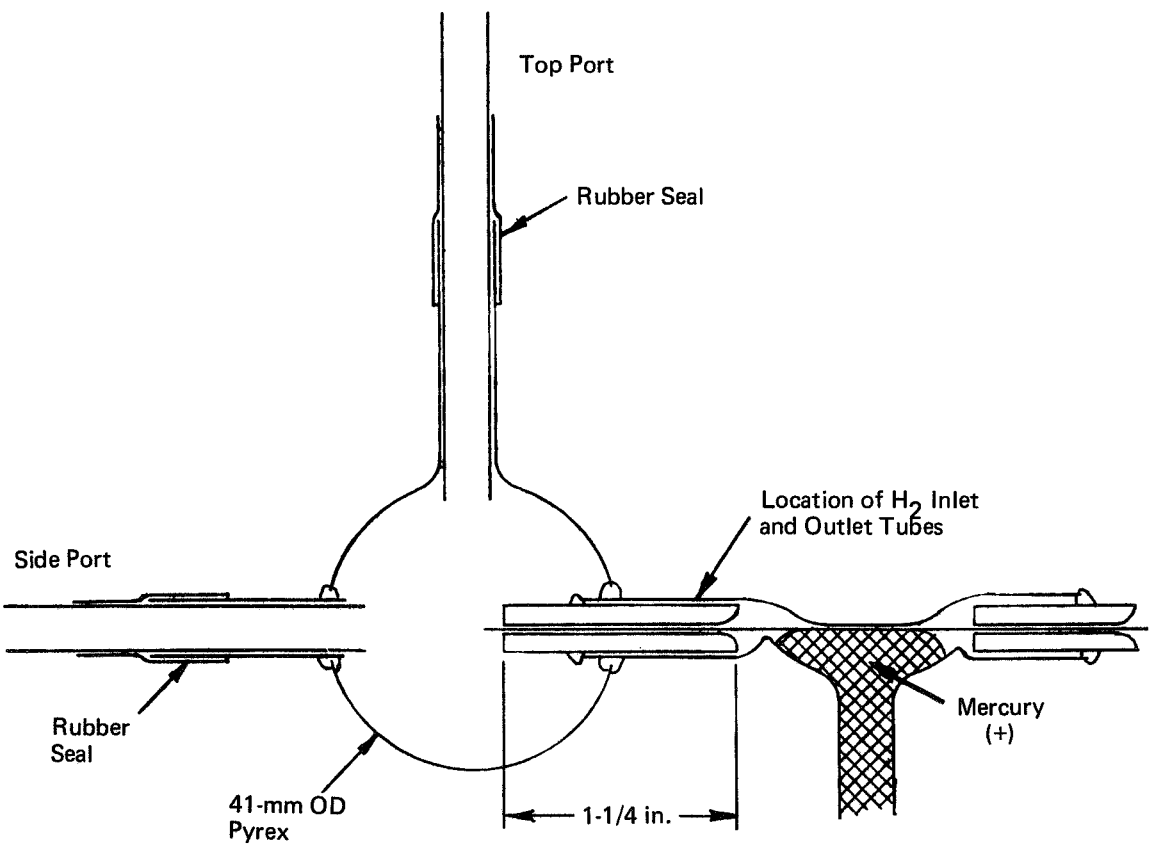
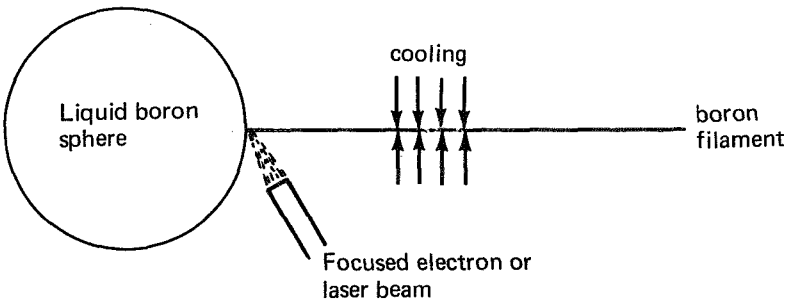
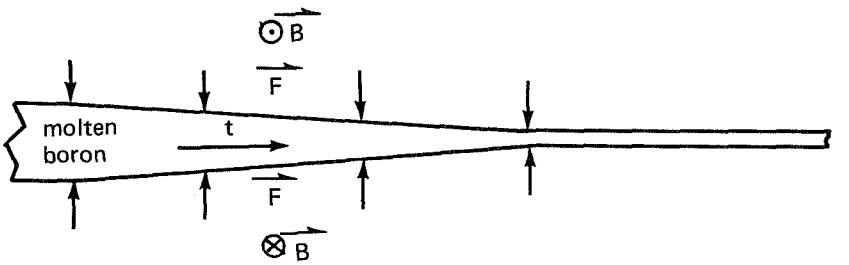


Figure 8. Glow Discharge Apparatus

- Point Destruction of Surface Tension



- Electrical Discharge Forming



- Melt Drawing

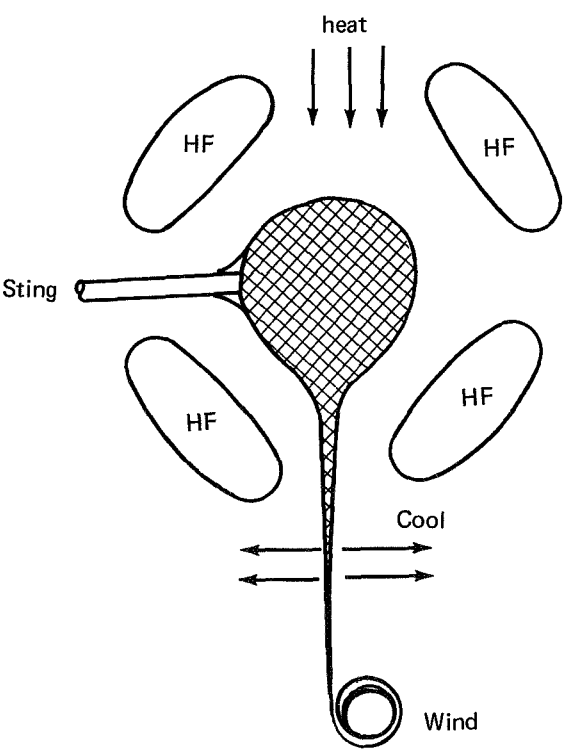
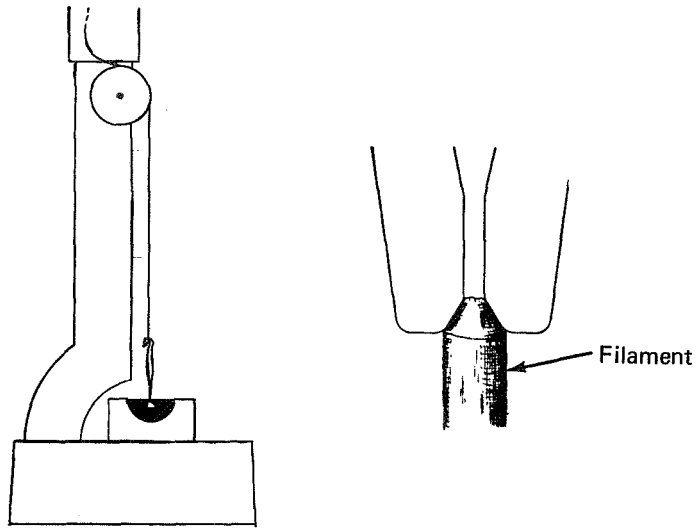
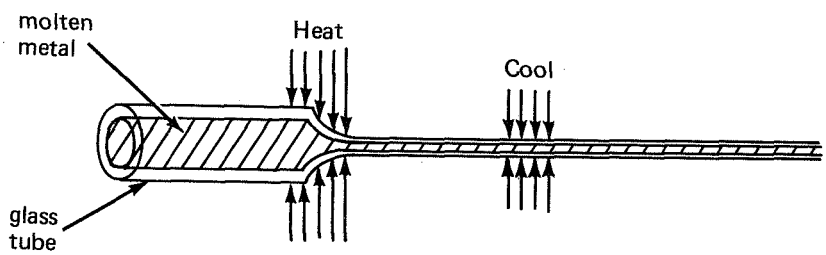


Figure 9. Point Destruction, Forming, and Drawing Processes From the Melt

- Czochralski Crystal Growth[56]



- Taylor Process



- Drawing of Substrate Through Melt[39]

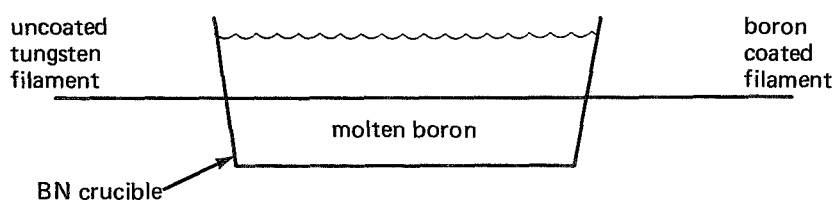


Figure 10. Czochralski, Taylor, and Substrate Drawing Processes From the Melt

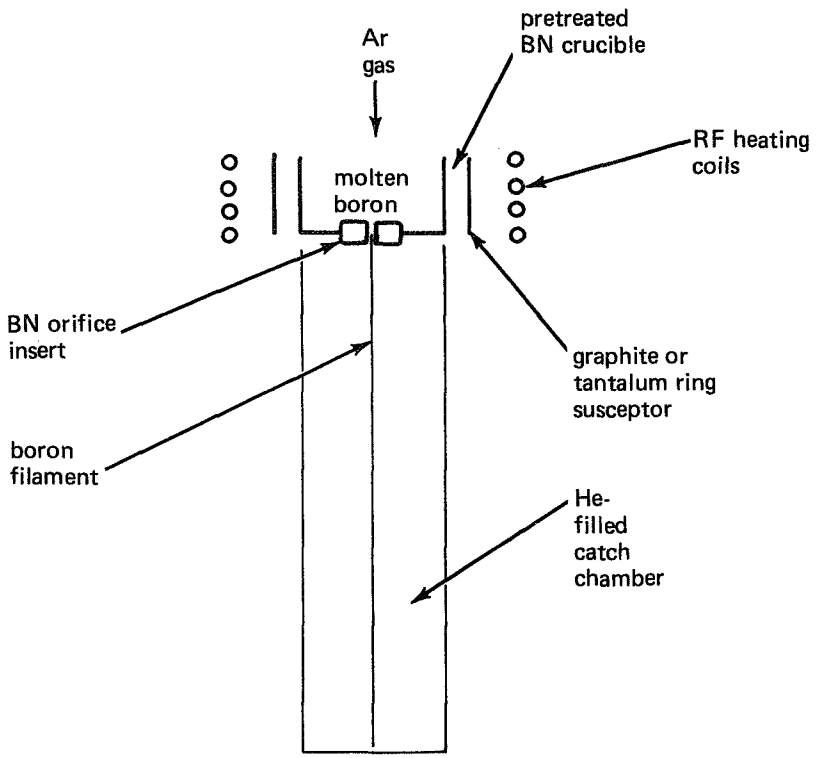


Figure 11. Melt Extrusion Apparatus

- Crucible and Orifice erosion
- Low filament strength due to surface pitting, grain size, and solidification mode
- Incomplete mixing of alloys
- Breakage upon impact in collection chamber

Figure 12. Problems with Melt Extrusion to Date

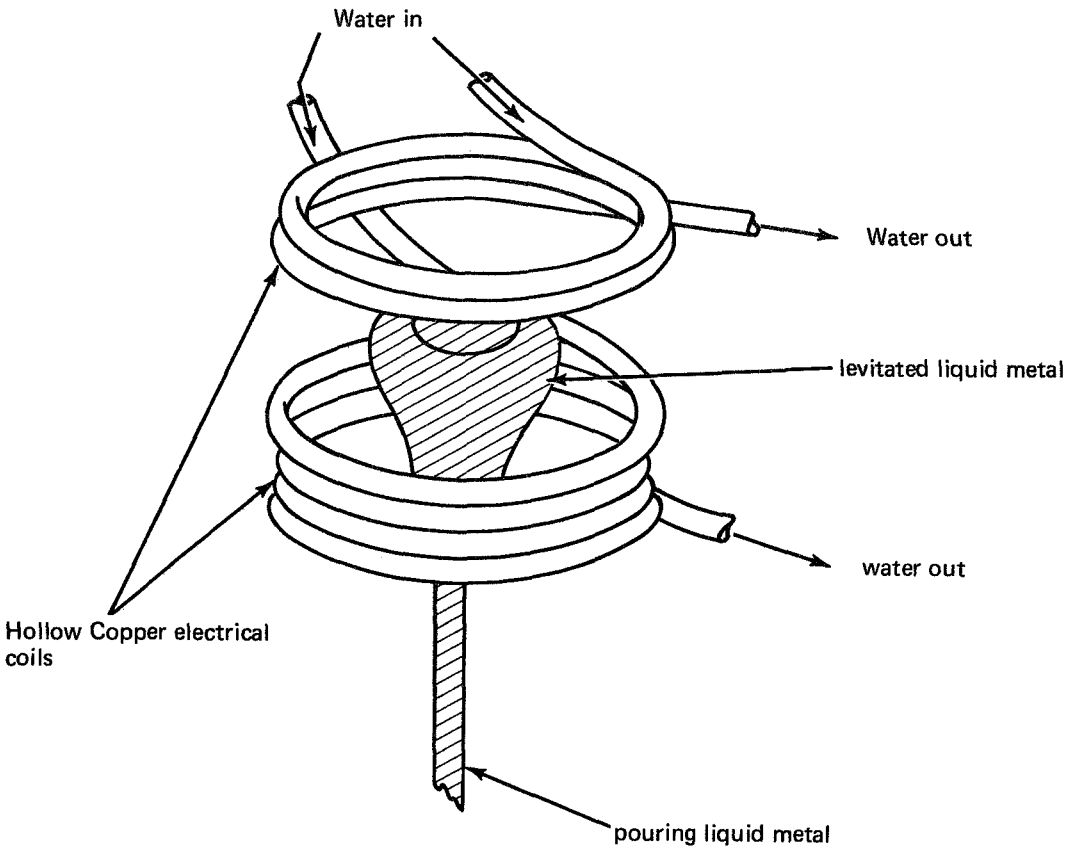


Figure 13. Levitation Melting Apparatus [52]

Ag	Cr	Ga	In	Nd	Pt	Sn	Y
Al	Cu	Gd	Li	Ni	Ru	Ta	Yb
Au	Dy	Ge	Mg	Pb	Sb	Ti	Zr
Be	Er	Hf	Mo	Pd	Si	U	
Co	Fe	Ho	Nb	Pr	Sm	V	

Figure 14. Known Levitation-Melted Elements [48]

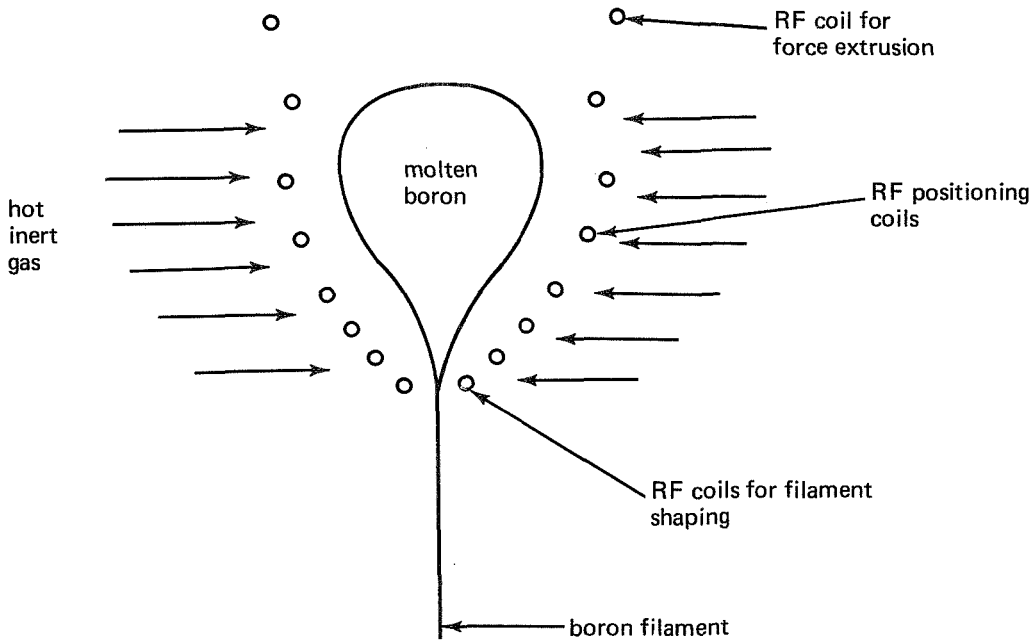


Figure 15. Boron Filament Manufacture in Space

- Lower Cost
- High Quality Filaments
- Faster Production Rate
- More Potential Uses

Figure 16. Summary of Proposed Process Advantages

- Solidification of boron
- Levitation, space positioning, and field extrusion of boron
- Mechanical properties of melt-produced boron
- Design of production machine
- Detailed cost analysis

Figure 17. Summary of Areas For Further Investigation